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Reduced Graphene Oxide nano-composites layer on fiber optic tip sensor reflectance response for sensing of aqueous ethanol

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Abstract

In this study, the used of tapered optical fiber tip as sensors coated with reduced Graphene Oxide (rGO) is investigated. The resultant rGO nanocomposites coated on the tapered fiber sensor were characterized by X-ray Diffraction (XRD), Raman spectroscopy, and field emission scanning electron microscopy (FESEM). Optimization of the rGO layer and the tapering parameters are found and the sensing capability of the device is tested using different concentrations of ethanol in water. The nanocomposite layer improved the performance of the sensor by demonstrating high sensitivity to aqueous ethanol when interrogated in the visible region using a spectrometer in the optical wavelength range of 500–700 nm. The reflectance response of the rGO coated fiber tip reduced linearly, upon exposure to ethanol concentrations ranging between 20-80 %.

Keywords: Reduced graphene oxide, Optical fiber sensor, Reflectance

Background

The use of optical fiber as a sensor gained much interest in the last decade. Optical fiber sensors have several advantages over electrical based sensors in many chemical and biological applications [1]. The most intriguing advantages lies in the miniaturization and response time. The development of submicron-sized optical fiber sensors is the technology based on nanofabricated optical fiber tips [2, 3]. Optical fiber sensors have been demonstrated in measuring the pH of buffer solutions inside micron-size holes in polycarbonate membrane [4]. These submicron pH sensors have millisecond response times due extremely small sizes.

The basis of fiber optic based sensors lies in the geometry of the fiber itself. Fiber optic is made of a plastic or glass core surrounded by a layer of cladding material [5]. The difference in density or refractive indices between these two materials enables the light propagation in an optical fiber in accordance with the principle of total internal reflection [6]. Optical fiber are mainly used as a sensors for physical changes, multimode fiber(MMF) can also be used to sense refractive index change but commonly involve tapered MMF and coating it with other materials to expose the core to the new surrounding area [7]. In this process the waist size of the MMF is reduced, to a point which all the core and cladding becomes a new core, so that the MMF area can be immersed in the sample and the sample can act as the new cladding to the fiber. Exclusively, the sensitivity of the sensor increase as cladding thickness decrease. This technique is particularly effective but it is more relevant to the change of the total internal reflection, TIR [6].

The optical and electronic properties of graphene attracts tremendous interest in the science of optical sensing. Graphene has high optical transparency and mobility [8]. In additiion, graphene characteristics are flexibility, robustness and environmental stability [9]. In particular, graphene oxide(GO) and reduced graphene oxide(rGO) have been used as an composite layer in energy storage denses [10], biomedical applications [11] and electronic components [12].

Al-Qazwini et al. [13] shares that the performance of an surface plasmon resonance (SPR) based optical fiber sensor using finite-difference time domain. The results show that the performance of the fiber sensor can be optimized by choosing a proper combination of metal layer thickness of 40–60 nm and residual

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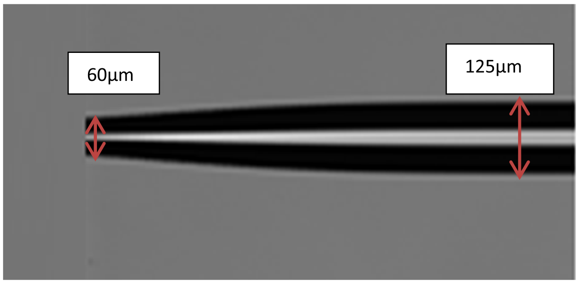

Fig. 1 Tapered fiber tip

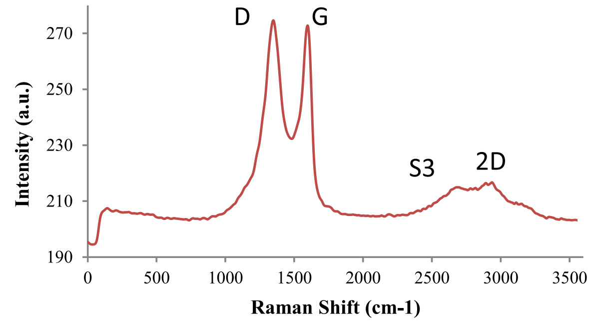

Fig. 3 Raman spectra of rGO thin film on glass substrate showing the D, G, S3 and 2D peaks

cladding thickness of 400–500 nm. In additon, they investigated an SPR-based optical fiber sensor by modeling a simple planar waveguide structure composed of four superimposed layers substituting the gold-coated polished single-mode optical fiber.

In this paper, we report a tapered optical fiber tip coated with rGO for sensing ethanol in water. Reduced graphene oxide is prepared from reduction of graphene oxide by thermal, chemical or electrical treatments [8]. Graphene film fabrication from solutions of GO have attracted considerable attention because these procedures are suitable for mass production. However, GO is an insulator, and therefore a reduction process is required to make the GO film conductive. rGO is used for the sensing nanocomposite layer due to its high ccnductivity [14] and the solubility in ethanol is higher [15] than GO compared to the work by Shabaneh et al. [16], rGO have much more simple fabrication process compared to GO. By varying the tapering profile, and improvisation of the rGO thickness, we enhanced the sensitivity of the sensor.

Methods

Multimode fiber (MMF) with a core and cladding diameter of 62.5 µm and 125 µm respectively was used in this research [17, 18]. Vytran glass processing workstation (GPX 3000 series) was used to taper the multimode fiber to obtain a waist diameter of 60 µm with waist length of taper is 5 mm, and for both downtaper and uptaper is 3 mm. After that, the tapered fiber will be cleaved in the middle as shown in Fig. 1, obtained from the camera of the Vytran workstation. It shows that the tapered tip has a diameter of 60 µm and has a smooth taper with a clean cleaved tip.

The fiber tips are then coated with different concentrations of RGO using drop casting method covered the tapered area. To do this, the fiber tips are placed in a 70 °C oven for 20 min to completely dry the tips and prepare the tips for the annealing process. Then 1 ml RGO of 0.2, 0.5, 0.75 and 1.0 mg/mol concentrations are dropped on each tip respectively. These fiber tips are then returned to the oven for annealing at 70 °C for 1 h.

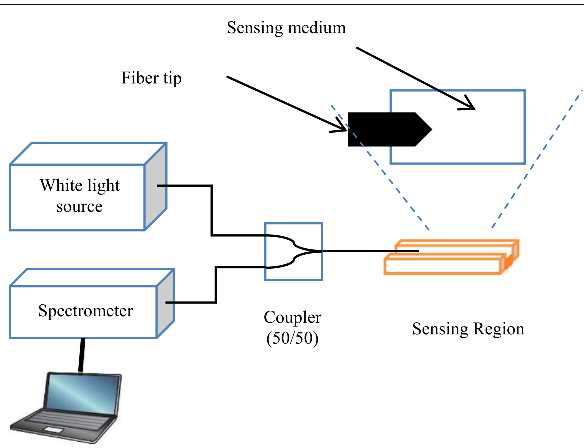

Fig. 2 Experimental Setup

The experimental setup of the project is shown in Fig. 2. Ocean optic whitelight source (HL 2000 ocean optics) and spectrophotometer (USB 4000 ocean optics) were used as the input and detector respectively. The fiber tip is placed in a flow cell and the reflection of the light from the tip is captured through a coupler. The spectraSuite software captures and presents the data in a graph.

Results and discussion

Figure 3 shows the Raman spectrum of the rGO thin film on glass substrates. The measurements are carried

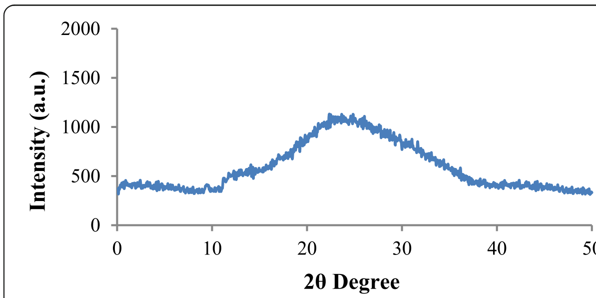

Fig 4 XRD of rGO thin film on glass subtrates

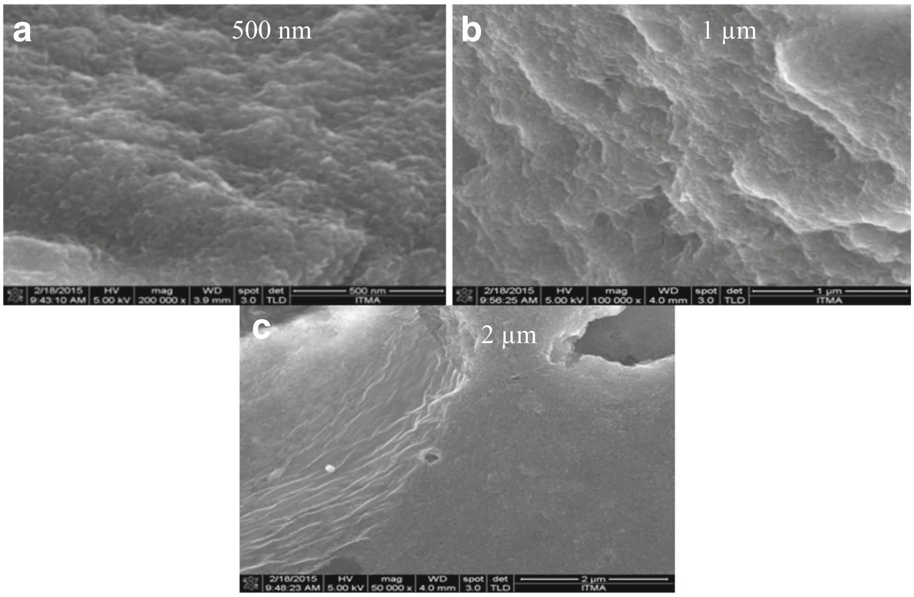

Fig. 5 FESEM image of reduced graphene oxide sheets on glass substrate at (**a**) 500 nm (**b**) 1 micron and (**c**) 2 microns scale

out by Raman spectrometer (Renishaw) using laser source with λ = 514 nm. The spectrum reveals the four characteristic D, G, 2D and S3 peaks of RGO. The D peak at about 1336 cm-1 generates from the breathing modes of six- membered rings that are activated via structural imperfections caused by the attachment of hydroxyl and epoxide groups on the carbon basal plane. The G peak at 1585 cm-1 duly corresponds to the first-order scattering of the E2g phonon mode at the Brillouin zone center. The 2D peak at 2831 cm-1 is the second order of the D peak and the S3 peak at 2585 cm-1 is due to the imperfect activated grouping of phonons.

XRD analysis of the rGO nanocomposites on the substrates is shown in Fig. 4. In this research, a broad peak is observed from 10 to 40° with the highest intensity at

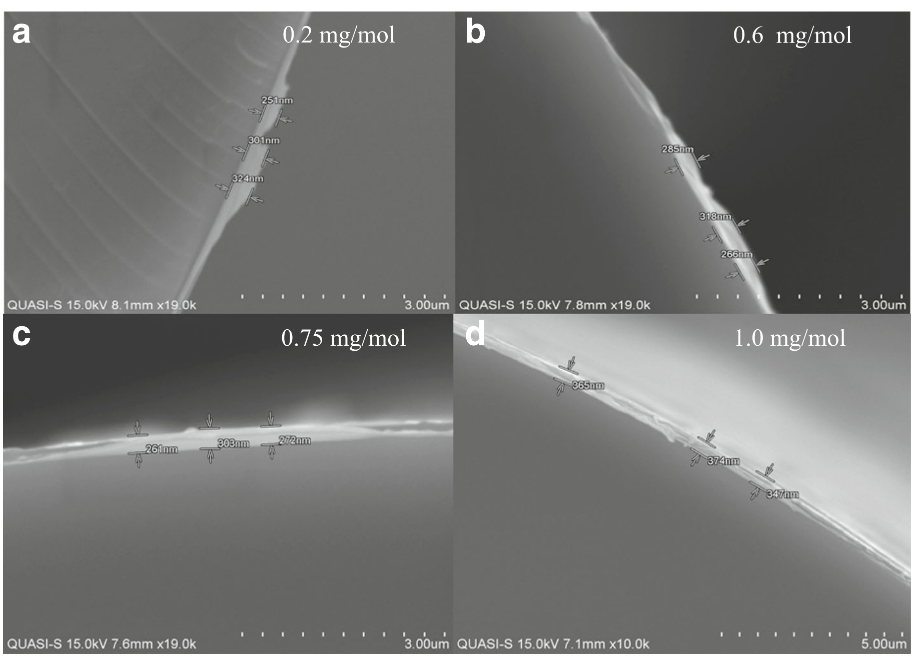

Fig. 6 Cross-sectional SEM image of reduced graphene oxide layers deposited on optical fiber tip for concentrations (**a**) 0.2 mg/mol (**b**) 0.6 mg/mol (**c**) 0.75 mg/mol (**d**) 1.0 mg/mol

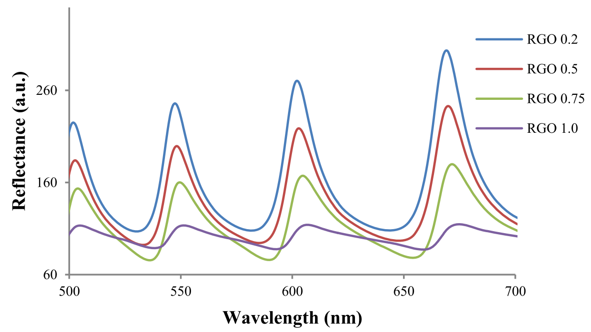

Fig. 7 Reflection spectrum of tapered fiber tip with different thickness of rGO

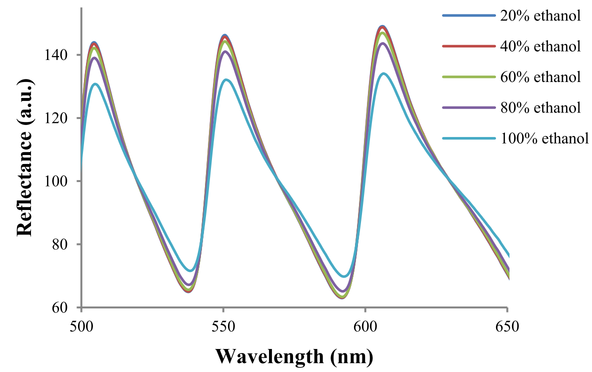

Fig. 9 Reflection spectrum of rGO (0.75 mg/mol) coated tapered fiber tip for different ethanol concentrations

23.65° which may be attributed to partial restacking of exfoliated graphene layers [19–21].

The morphology of the rGO nanocomposites is observed by field emission scanning electron microscopy (FESEM) and scanning electron microscopy(SEM). FESEM image is obtained using FEI Nova Nano SEM400 with 5.0 kV source. Figure 5a to c shows the FESEM image of the rGO nanocomposites. It reveals that the rGO nanocomposites are in the form of nano sheets.

Figure 6a to d show the SEM images of the cross section of the fiber tip showing the thickness of the rGO layers with concentrations of 0.2, 0.5, 0.75, 1.0 mg/mol The average thickness of the rGO layer with concentrations 0.2, 0.5, 0.75, 1.0 mg/mol is 292 nm, 290 nm, 279 nm and 362 nm respectively.

The folding of the rGO sheets contributes to a darker shade on the SEM image. This implies that the graphene oxide is single to a few layers thick. Furthermore, the rGO sheets adhered well to the substrate, promoting reflectance on the rGO nanocomposites. The thickness of the nano structured rGO nanocomposites is estimated to be approximately 20–30 mm, due to the overlapping of the nanosheets. This SEM is performed to verify the uniformity of the coating of rGO films on the substrates. These micro characterization results are significant to verify the morphology of the rGO nanostructured thin films.

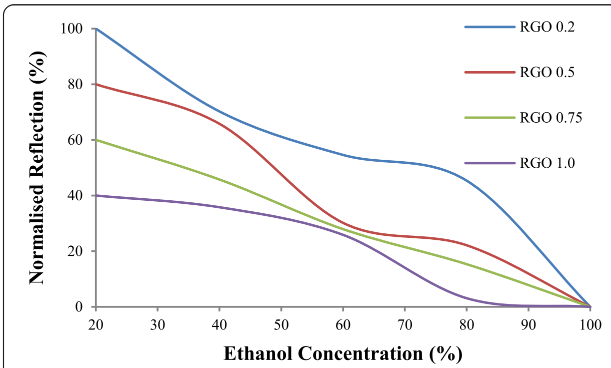

Fig. 8 Comparison of normalized reflection of rGO coated tapered fiber tips for different concentrations of ethanol

The reflectance spectrums of the fiber optic tips with different concentrations of rGO are investigated. As shown in Fig. 7, the reflectance decreases as the concentration of the rGO is increased. This is due to higher absorbance of the light signal as the thickness of the rGO layer increases. The interaction between the different concentrations of ethanol molecules and rGO on the tapered fiber tip transforms the optical characteristic of the rGO films, resulting in the proportional response of the developed sensors towards ethanol [6].

In order to find the optimum thickness for rGo nanocomposites the fiber optic tips are tested with 5 concentrations of ethanol, from 20 to 100 %. The results are summarized in Fig. 8. For all the rGO coated tips, the intensity drops as the ethanol concentration increases. The fiber optic tip with the 0.75 mg/mol tip gave the stable response for all wavelengths. Interference are specially required the changes of parameters such as taper waist diameter, length, and transition form [7, 22, 23].

The reflectance spectrum and dynamic response of the sensor is investigated for the 0.2 mg/mol, 0.5 mg/mol, 0.75 mg/mol and 1.0 mg/mol rGO coated tip at a wavelength range of 500 to 650 nm. The results show that at 0.75 mg/mol rGO, that intensity decreases as the concentration of the ethanol is increased as shown in Fig. 9. Table 1 shows the percentage different between difference ethanol concentration in water. From Table 1, it

Table 1 Sensitivity for different concentration of ethanol

Ethanol	Δ_{ref}	Δ_{r1} - Δ_{r2}
20 %	a_1	-
40 %	a_2	0.349
60 %	a_3	1.77
80 %	a_4	3.336
100 %	a_5	9.645

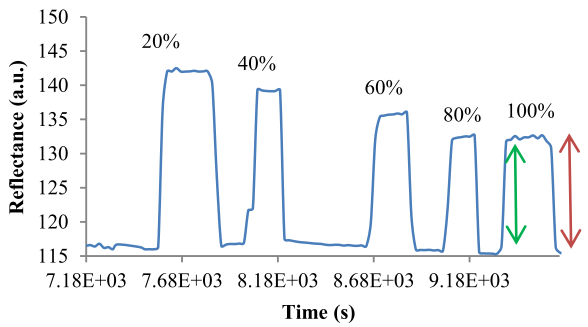

Fig. 10 Dynamic response of RGO (0.75) coated tapered fiber tip. (green = response, red = recovery)

can be conclude that sensitivity for the developed sensor is approximately 3.0196.

For the fiber tip with 0.75 mg/mol rGO concentration, the dynamic response is presented in Fig. 10. It shows that the higher the concentration of ethanol, the higher the reflectance response of the sensor. The sensor shows high sensitivity that can be observed from the dynamic response of the reflectance plotted. For every ethanol concentration, the sensor responds to a stable level and when the ethanol is removed, the sensor recovers and returns to a stable baseline. The response and recovery time was obtained as 40 and 70 s respectively.

Conclusion

The performance of an ethanol sensor using a tapered fiber tip coated with a new material, rGO was investigated in this research. We have successfully designed and fabricated a tapered fiber tip optic sensor coated with reduced graphene oxide (rGO) as new sensing layer to detect different concentrations of ethanol in water.

The results show that this enhancement of the sensing surface is able to deliver high sensitivity for the detection of various concentration of aqueous ethanol. The tapered fiber optic tip also gives a stable repeatable response towards ethanol concentrations as well as fast response and recovery time of 40 and 70 s respectively. Moreover the introduction of the layer of rGO nano-particle also helps increase its structural strength. The experimental data also determines that the thickness of the rGO layer to produce the optimal results is approximately 280 nm.

It show that fiber tip optic with a higher concentration rGO which mean of higher refractive index coating are more sensitive compared to the lower concentration rGO-coated fiber tip.

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Authors' contributions

MAAR: Sensing experiments. PTA: Material characterization. HNL & NMH: GO synthesis. ASMN: Data analysis. All authors read and approved the final manuscript.

Competing interests

The authors declare that they have no competing interests.

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